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High-Temperature Measurements

AS in other fields of research, progress in the high-temperature program of the National Bureau of Standards has depended largely on the development of suitable instrumentation. In response to needs in critical areas of technology and in fundamental physics and chemistry, improvements have been made in many of the methods for measuring and generating high temperatures and for determining physical and chemical properties of materials at those temperatures.

The basic instruments for measuring temperatures above 1,000°C are the optical pyrometers that are calibrated at the Bureau by comparison with its standard pyrometer. This, in turn, is calibrated by observing a blackbody maintained at the freezing point of gold (1,063°C), one of the primary fixed points on the International Temperature Scale. The Bureau is also responsible for improving the accuracy of this scale; a recent development is a photoelectric pyrometer that eliminates personal error in making the brightness match that is part of the pyrometer procedure.

Although the processes taking place, for example, in the combustion chamber of a rocket engine must often

be studied under actual operating conditions, the use of such a source of heat for laboratory investigations of fundamental properties of materials is generally ruled out by difficulties in controlling the temperature and in isolating the process studied from the effects of extraneous reactions. Instead, various other types of heat source have been developed—electric arcs, solar furnaces, resistance and induction electric furnaces—each with its particular advantages for special kinds of high-temperature research. Improvements in design or method of use have been made in nearly all of these devices.

Considerable progress has also been made in developing or improving instrumentation for measuring crystallographic, thermodynamic, and chemical properties of materials at elevated temperatures. Included are instruments used in investigations of inversions and phase transitions by differential thermal and thermogravimetric analysis, in X-ray diffraction studies of crystal structure and its changes at high temperatures, in observation of gas-solid reactions by time-lapse photography, and in measurement of liquid viscosity.

HIGH-TEMPERATURE MEASUREMENTS ISSUE

The second of two consecutive issues devoted to high-temperature research, this issue contains articles relating to measurement problems at high temperatures. The June issue dealt with high-temperature structure, properties, and processes.

Photoelectric Pyrometer

During the past year the high-temperature measurements laboratory constructed a photoelectric pyrometer¹ that is expected to replace the presently used disappearing-filament optical pyrometer for measuring temperatures above 1,000° C. The basic optical system and disappearing-filament principle are retained, but the brightness of the test body is compared with that of the pyrometer lamp photoelectrically rather than visually. Early tests of the photoelectric device show a substantial gain in precision.

The instrument consists of a telescope with a 2.5-in. objective lens that is sighted on the test object whose temperature is to be measured. A lamp with a single horizontal filament stands inside the telescope on the optic axis, and the image of the test object is focused in the plane of the filament. An eyepiece lens then forms an image of the filament superimposed on the image of the test object. To the eye, the filament



Measuring the temperature of a lamp (right) with the photoelectric pyrometer recently designed and constructed by the Bureau for temperatures above 1,000° C.

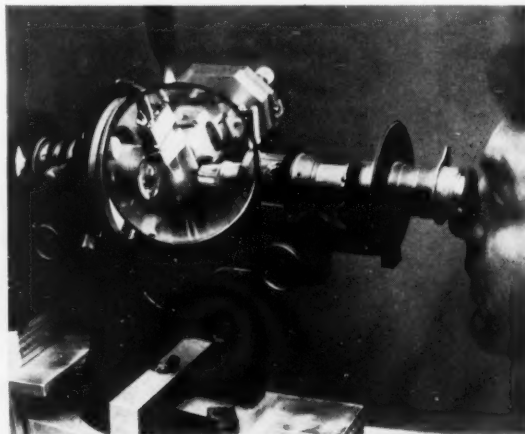
can be made to disappear against the background of light from the test object by suitably adjusting the current in the filament. The relation between current, brightness, and temperature of the filament is determined from observations on a blackbody at the gold point (1,063° C) and calculations based on the fundamental laws of radiation.

In order to replace the visual judgment of equality of brightness by a photoelectric determination, a diaphragm with a horizontal slit is set in the image plane of the eyepiece lens and a photomultiplier tube behind the slit measures the intensity of the light passing through. A rectangular block of glass, with optically flat faces and about 1 in. thick, is placed in front of the slit and made to rotate slowly. This causes the image (filament against background of test object) to move past the slit, so that successive portions of the

image are scanned by the photomultiplier tube. If there is no variation in brightness as the image is scanned, no change in photomultiplier current is observed.

In this way a precision of 0.1° C is attainable when observing a blackbody at 1,100° C. An interference filter with a bandpass of roughly 50 Å (angstroms) can be placed before the slit to make the instrument approximately monochromatic; most tests have been made in this manner, in the vicinity of 6500 Å.

The detector is a commercially manufactured experimental photomultiplier tube using the recently discovered multi-alkali metal cathode which has an extremely high quantum efficiency (ranging from 19% at 4200 Å to 4% at 6500 Å). The cathode consists of a mixture of all five of the alkali metals, and the reason for its unusual effectiveness is not definitely known.



Hot-wire furnace (seen through magnifying glass) in apparatus for growing small crystals at high temperatures.

Electric Furnaces

Though other methods of generating high temperatures have been developed (e. g., solar furnaces, electric arcs, high-velocity gases), the electric furnace—resistance or induction—continues as a major research tool for temperatures as high as 3,000° K. In the Bureau's high-temperature program a very large number of electric furnaces are employed. To meet the requirements of advanced research, many of these have been adapted to new experimental techniques and improved designs have been developed that extend the upper limit of temperature attainable or produce more uniform and accurately determined temperatures.

For investigations in air or other oxidizing atmospheres, conventional wire-wound resistance furnaces are restricted to a maximum of 1,900° K, the highest temperature at which platinum can be used with any dependability. This upper limit is increased to 2,300° K in the thoria (thorium oxide) resistance furnace developed by the Bureau.² In this furnace, the thoria heat-



Crystal growth at $1,400^{\circ}\text{C}$ is observed through a microscope.

ing elements contain a small amount of cerium oxide to increase their conductance. The conductance is still so low at moderate temperatures that it is necessary to preheat the thorium with auxiliary heaters to $1,200^{\circ}\text{K}$, at which temperature, however, the resistance is reduced sufficiently for the thorium to carry its own heating current. Because of its large capacity and its applicability to work under oxidizing conditions, the thorium furnace has been particularly useful in determining melting points, coefficients of thermal expansion, and phase equilibrium relationships of refractory materials.

Temperatures above $1,900^{\circ}\text{K}$ are usually obtained with molybdenum, tungsten, tantalum, and graphite heating elements. These substances must be protected against oxidation by a neutral or reducing atmosphere, and this seriously restricts the atmospheric conditions in which investigations can be undertaken. Nevertheless, within these conditions, the Bureau has many inductive and resistance furnaces capable of reaching $3,000^{\circ}\text{K}$ and higher.

One such furnace, capable of attaining $3,300^{\circ}\text{K}$, incorporates a hydraulic press for applying pressures of several thousand pounds per square inch to small samples.³ The heating element is a long graphite tube which is operated in a protective atmosphere of helium or argon. For thermal insulation, the graphite tube is embedded in carbon black and the assembly is sealed to prevent seepage of gas when the hydraulic press is used. Refractory samples to be studied under pressure are encased in graphite molds (dies) and mounted inside the heater tube which is held vertically between the plates of the press. Under pressure, powdered samples can be formed into dense, predetermined shapes; without pressure, a large number of phase equilibria and rates of reaction among mineralogical and metallurgical materials can be studied.

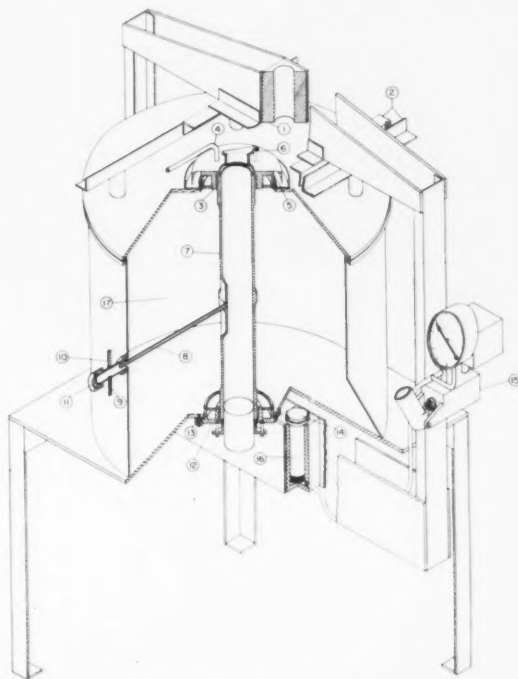
Among other special-purpose furnaces are a crystal-growing apparatus using a hot-wire type of furnace, and a high-temperature centrifuge.⁴ The first of these was developed as a result of unsuccessful attempts to make crystals of tricalcium aluminate for X-ray diffraction by conventional methods. It was concluded

that crystals of the desired small size could best be grown from a relatively small volume of melt. The crystals are propagated at the point of a V-shaped loop of fine platinum-rhodium wire mounted under a stereoscopic microscope. As crystallization progresses, the temperature within the droplet of melt can be measured continuously and the crystal growth visually observed.

The apparatus for high-temperature centrifugation of refractories consists of a platinum-wound vertical tube furnace below which is mounted a motor designed to turn at 17,000 rpm. The device maintains samples at $1,350^{\circ}\text{K}$ in a centrifugal field several hundred times that of gravity.

Solar Furnace

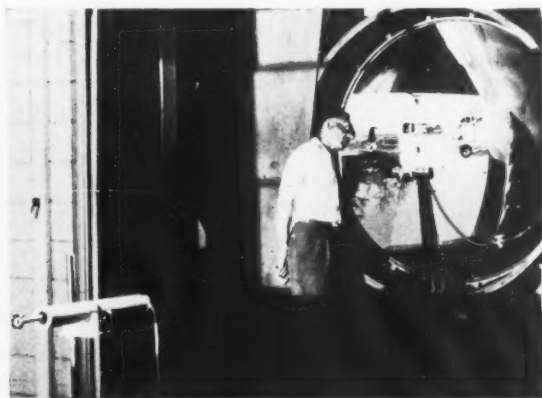
One of the more recent additions to the Bureau's high-temperature research facilities is a solar furnace, a device that concentrates the rays of the sun onto a small area where materials can be raised to tempera-



Cut-away drawing of graphite-tube resistance furnace incorporating a hydraulic press. 1, Upper platen of press; 2, press carriage and rail; 3, upper brass water-cooled terminal; 4, copper water-tube inlet and bus bar (outlet and bus terminal not shown); 5, zircon sand expansion seal; 6, flange for asbestos seal; 7, 3-in.-diam graphite tube heater element; 8, graphite sighting tube; 9, inert gas inlet; 10, pyrometer support; 11, lucite window; 12, lower water-cooled terminal; 13, transite insulator; 14, lower platen of press; 15, hydraulic band pump; 16, hydraulic ram; and 17, carbon powder insulation.

tures up to 3,500° C, or about two-thirds the temperature of the sun's surface. Thus far it has been used principally for studies on the vaporization of refractories—materials that can withstand prolonged exposure to high temperatures. Experiments are also being conducted on the use of the solar furnace for the zone-melting method of refining oxides of zirconium, thorium, and uranium. Eventually it may be possible to grow single crystals of these and similar materials for laboratory studies.

For studies of refractories, the tungsten arc, carbon resistor, and electric induction furnaces have the drawback that the electrodes, resistors, or crucibles contaminate the reaction and thus lead to confused results. Because of its "clean" source of heat, the solar furnace avoids these difficulties. In studying the vaporization



Parabolic mirror of solar furnace. Near center of mirror is a reduced, inverted image of the 3-ft-square heliostat mirror that is driven by motor so it constantly reflects sunlight into the fixed parabolic mirror. Photo-cell assembly that controls motor is at bottom left.

of refractories the materials can be evaporated directly from a hot spot of known diameter (0.25 in.) to the cool surface of the glass bulb containing the specimen. Furthermore, the atmosphere over the hot spot can be changed without introducing unwanted reactions. On the other hand, there is serious difficulty in accurately measuring the temperature.

The Bureau's solar furnace was converted from a surplus Army searchlight with a parabolic mirror 5 ft in diameter. The spherical glass around the specimen is placed so that the latter is at the focus of the mirror; the glass itself remains cool because the image of the sun is unfocused where the light passes through. Light from the sun is first caught by a heliostat, a motor-driven 3-ft-square plane mirror; by means of an electronic control circuit guided by photocells, the plane mirror is turned so that it follows the apparent motion of the sun across the sky and constantly reflects the light towards the fixed parabolic mirror.

Electric Arcs

The highest sustainable temperatures available in the laboratory are those within the core of electric arcs—values as high as 50,000° K have been reported. One of the newer Bureau programs, sponsored by the Air Research and Development Command, is investigating the processes that take place within such arcs in order to obtain data on chemical equilibria and thermodynamic properties of gases at high temperatures. The work thus far has dealt primarily with arcs of 40- to 200-amp current which yield temperatures from 10,000° to 20,000° K and can be operated steadily for periods of 10 min or more.

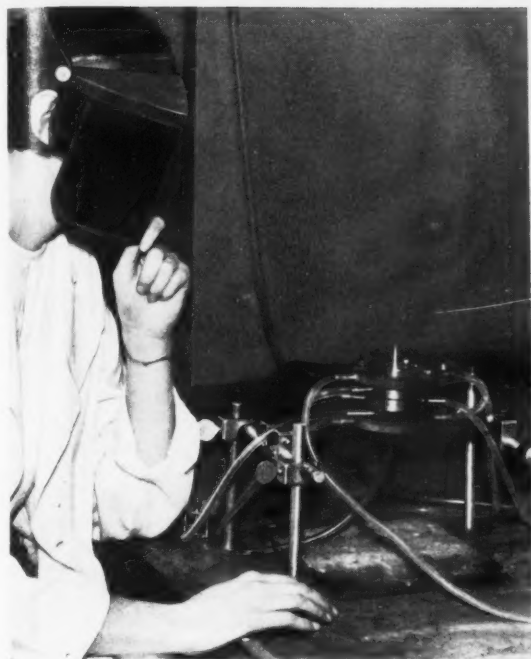
In spite of the very complicated detailed mechanism for the conduction of current and heat in an arc, the gas actually exhibits local thermodynamic equilibrium everywhere except in the immediate vicinity of the electrode surfaces. As a result, it is possible to calculate the gas temperatures in the usual sense of the word from gas compositions as determined by spectrographic measurements. Given a complete set of data of this kind over the range of temperatures found in



Electric arc apparatus for studying processes in gases at temperatures up to 10,000° C. Scientist is lowering the upper electrode to initiate the arc.

the cross section of an electric arc, one can then calculate many important thermodynamic quantities for gaseous mixtures at the high temperatures of the arc.

Up to the present the work has consisted mainly in the measurement of temperature profiles of arcs in simple pure gases. It is planned next to introduce various solid, liquid, and gaseous materials into the arcs and to study the resulting chemical equilibria by means of composition and temperature measurements. It is further planned to obtain transport properties such as diffusion coefficients and thermal and electrical conductivities of gases at the high temperatures prevailing within the arc from data on temperature, composition, current, and voltage. These transport data should give some insight into the effective laws of force between particles in excited energy states.



Experimental electric arc generator for high-temperature research.

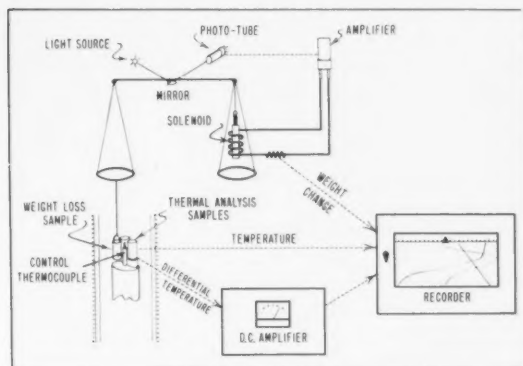
Differential Thermal Analysis and Thermogravimetric Analysis

Some reactions in solids at high temperatures can be studied by quenching, or cooling very rapidly, to room temperature, and then studying the nature and extent of the reaction in the cooled sample. However, for substances in which reversible transitions or phase changes occur, or in which reactions occur on cooling that cause the sample to disintegrate, the quenching method cannot be used. In such cases the desired information may be obtained by the method of differential thermal analysis. In principle, this consists in heating the sample at a carefully controlled rate and observing the slight deviations from the impressed heating rate due to reactions that absorb or evolve heat. Information about the reactions taking place can then be obtained from the known relations that connect the temperature distribution in the reacting sample, reaction rate, heat of reaction, and thermal diffusivity of the sample. This method has been used to study reversible transitions in solid solutions of the divalent carbonates at elevated temperatures. It has also been applied to oxide systems and mineral polymorphism.

If a gaseous phase is involved in a solid-state reaction, the sample weight changes as the reaction proceeds. To study reactions of this type, an apparatus for automatically recording weight changes as a function of time and temperature has been developed.

Although limited to a maximum temperature of $1,250^{\circ}\text{C}$, this apparatus has been extensively employed in studies of the high-temperature behavior of natural and synthetic minerals—oxides, carbonates, phosphates, and others.

The thermogravimetric apparatus is basically a conventional laboratory balance modified so that changes of weight are balanced by adjustment of a magnetizing current. The balancing force results from the interaction of the magnetic field of a solenoid with the field of a permanent bar magnet suspended, inside the solenoid, from one side of the balance. Automatic balancing is achieved by means of a photoelectric sensing arrangement in which a beam of light is reflected to a dual phototube from a mirror mounted on the balance beam. The two sections of the phototube are connected in a bridge circuit, the output of which is amplified and applied to the solenoid. Any change in weight tending to produce unbalance is promptly counteracted by whatever change of solenoid current is needed to



Pictorial diagram of apparatus for simultaneous thermogravimetric and differential thermal analysis of materials subjected to high temperatures.

maintain balance (within a small fraction of a division of the balance scale). An electronic damping circuit eliminates oscillations and enables the electrical output of the instrument to follow faithfully very rapid changes of weight.

Because of the special advantages of combining the results of thermogravimetric analysis with those of differential thermal analysis, the two sets of equipment have been fitted into a unified arrangement. The output is on a single recording chart which simultaneously exhibits three quantities plotted against time—temperature, change in weight, and the differential temperature.

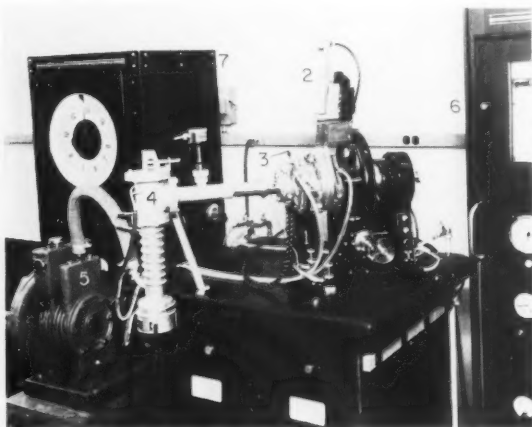
X-ray Diffraction Studies

X-ray diffraction is the principal technique for obtaining detailed information about the arrangement of atoms in a solid. No two compounds have atoms of the same size arranged in the same way, so that the X-ray

diffraction pattern of each compound is characteristic of that compound alone. The spacing between layers of atoms can be measured with an accuracy of 1 part in 10,000 and there is hardly a change, either in composition or spatial arrangement, that is too subtle to be detected.

Thus, by observing the X-ray diffraction pattern of a sample while heating it in a furnace, information is obtained that is useful for: (1) Identifying the crystalline phases by comparing with patterns of known phases; (2) detecting changes in phase caused by heat or chemical reaction; (3) measuring the very slight changes in spacing between planes of atoms caused by changes in composition (solid solution formation), thermal expansion, or mechanical stress; and (4) determining the exact arrangement and spacing of the atoms at each temperature.

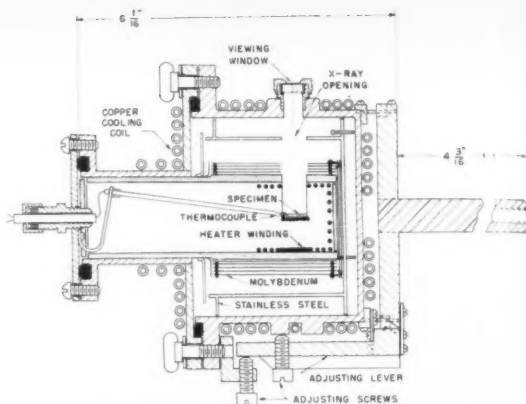
In 1946, very soon after Geiger-counter equipment for detecting diffraction patterns was introduced, the Bureau developed a high-temperature attachment that permits nearly continuous observation of the X-ray



Apparatus for X-ray diffraction studies at high temperature. 1, X-ray source; 2, X-ray detector (rotates about horizontal axis); 3, furnace containing specimen; 4, diffusion pump; 5, mechanical vacuum pump; 6, X-ray diffraction pattern recorder; and 7, temperature controller. See diagram of furnace, top right of page.

pattern while the temperature or atmosphere of the sample is being varied.⁵ With the new equipment and the high-temperature attachment, studies that formerly required a week can be made in a day; and transient effects too rapid to be followed by successive photographic exposures (the method previously used) can be studied by scanning a portion of the pattern as frequently as every 2 min. Melting, volatilization, chemical reactions, or deposition from the vapor phase can be observed conveniently by scanning back and forth over a single diffraction maximum characteristic of the phase of interest.

One series of Bureau studies utilizing these techniques began with a study of the oxides of manganese. This



Section through high-temperature furnace for X-ray diffraction studies. The X-ray opening extends around the specimen through an angle of about 180°; opposite the opening, in the outer wall, is a beryllium strip (not shown) which is vacuum tight but transparent to X-rays.

work was part of a study of fundamental factors affecting the performance of dry batteries. Studies of manganese oxides were then extended to include solid solutions of Fe_2O_3 and Mn_2O_4 . In another investigation the thermal decomposition of carbonates heated in various atmospheres was studied with the X-ray furnace and the results were correlated with data obtained by thermogravimetric analysis.

More recently, in a project sponsored by Wright Air Development Center, improved apparatus for accurate measurement of lattice parameters was designed and has been used to measure the thermal expansion of a number of materials being considered for high-temperature applications.⁶ The use of the X-ray method for this purpose has the advantage of requiring only 2 or 3 g of powdered material. From such a sample, accurate measurements of the thermal expansion in various directions in the crystal can be obtained. The type of apparatus employed in these studies, with the design improvements developed by the Bureau, is now being used in many other laboratories where high-temperature work is done.

¹ Photoelectric pyrometer, summary of paper by C. D. Johnson, Jr., presented at 42d annual meeting of the Optical Society of America, *J. Opt. Soc. Am.* **47**, 1058 (1957).

² Construction and operation of thoria-resistor-type furnaces, by S. L. Lang and R. F. Geller, *J. Am. Ceram. Soc.* **34**, 193 (1951).

³ Two resistance-type hot-pressing furnaces for laboratory use, by R. F. Walker and S. G. Bauer, *Rev. Sci. Instr.* **28**, 563 (1957).

⁴ Both of these are described in New techniques for new approaches to cement chemistry, by F. Ordway and T. F. Newkirk, *Ceram. Age* **58**, 28 (Nov. 1951).

⁵ High-temperature X-ray diffraction apparatus, by A. Van Valkenburg Jr. and H. F. McMurdie, *J. Research NBS* **38**, 415 (1947) RP1782.

⁶ Thermal expansion of cermet components by high-temperature X-ray diffraction, *Wright Air Development Center Technical Report* 55-473 (1955); a supplement to this is now in press.

Energy Distributions and Temperature Determinations in Flames

THE Bureau is conducting a program of basic research, sponsored by the Air Research and Development Command, on the physical and chemical processes occurring in flames and other high-temperature gases. In this work spectroscopic methods are employed to observe hot gases in both flames and electrical discharges. The data thus obtained are proving useful not only in reaching a better understanding of the combustion process but also in the development of accurate spectroscopic methods for measuring high temperatures. In addition, the program provides information on atoms and free radicals which is applicable to upper-atmosphere chemistry and physics, and chemical kinetics.

When present methods of temperature measurement are applied to certain systems of hot gases, several different temperatures are obtained. These temperatures must be related to the Thermodynamic or International Temperature Scales before valid thermodynamic calculations are possible. Detailed spectroscopic studies on well-controlled systems are thus needed to provide the basic information required in this area.

In measuring flame temperatures spectroscopically, molecular distributions are calculated from observed spectral intensities and compared with distributions predicted theoretically for certain temperatures. For the distribution calculations, the relative intensities of a group of related spectral lines emitted or absorbed by the flame are first measured. Then, from these relative intensity values and the known values for the probabilities of emission or absorption of radiation, the numbers of molecules in the various energy levels giving rise to spectral lines are determined.

The experimentally observed distribution may also be compared with distributions predicted theoretically for a range of equilibrium temperature values. If a certain temperature can be found that has a theoretical distribution agreeing with the observed one, then this is the temperature associated with the levels under study; or when the flame is substantially in equilibrium, this temperature is the temperature of the flame as a whole. However, the observed distribution does not always correspond to any temperature value. Even if it does, a parallel investigation of another group of



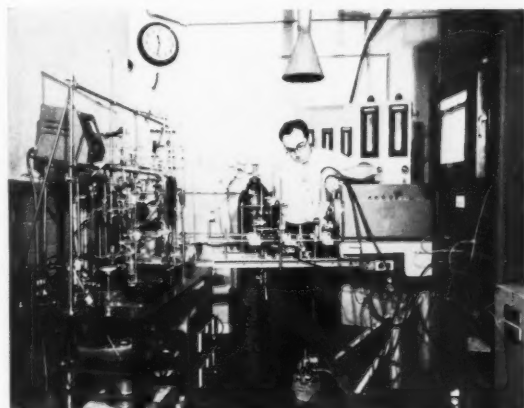
Spectroscopic measurements of radiation temperature included studies of flames similar to this acetylene-oxygen flame on a 64-mm-diam burner at 2 mm of Hg pressure.

energy levels in the same system may give a different temperature.

In using the observed distribution of molecules in the possible energy states to study rate processes and equilibrium conditions, three general types of behavior are found. Essentially they differ in the amount of interaction, or coupling, between the various groups of energy states in the system being observed. Examples of each type are discussed below.

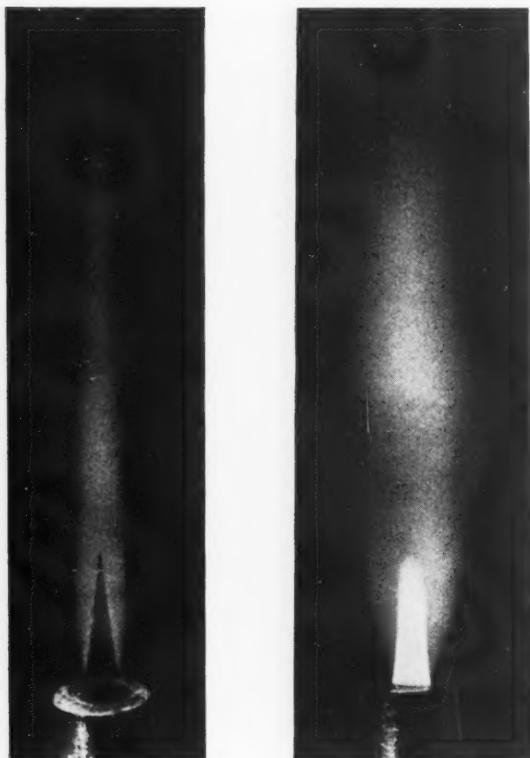
Strong Interaction

The simplest case is complete equilibrium. Here all the observed energy levels correspond to the same temperature so that the measurements constitute a reliable temperature determination. An example of this situation is a recent investigation of the emission spectrum of iron atoms added to a hydrogen-oxygen flame.¹ Here the intensity distribution in the emission spectrum corresponds to a temperature of 2,845° K. The temperature determined from the emission spectra of OH radicals in the flame is 2,770° K, which is also the



Apparatus used in spectroscopic studies of the temperature and composition of flames. This work is part of a search for fundamental information on the nature of combustion.

temperature determined from the corresponding absorption measurements. The temperature calculated from the known energy release in the flame is $3,100^{\circ}\text{K}$. The approximate agreement of these values indicates that the flame is essentially in equilibrium, that the several groups of energy levels have interacted strongly enough to come to the same temperature. Here the observed distributions do not depend on the details of the excitation process.



In studies of the combustion process, the hydrogen-oxygen flame (left) and acetylene-oxygen flame (right) were observed at atmospheric pressure. The bright inner cone of the acetylene flame is due to excitation of abnormally large amounts of free radicals containing carbon.

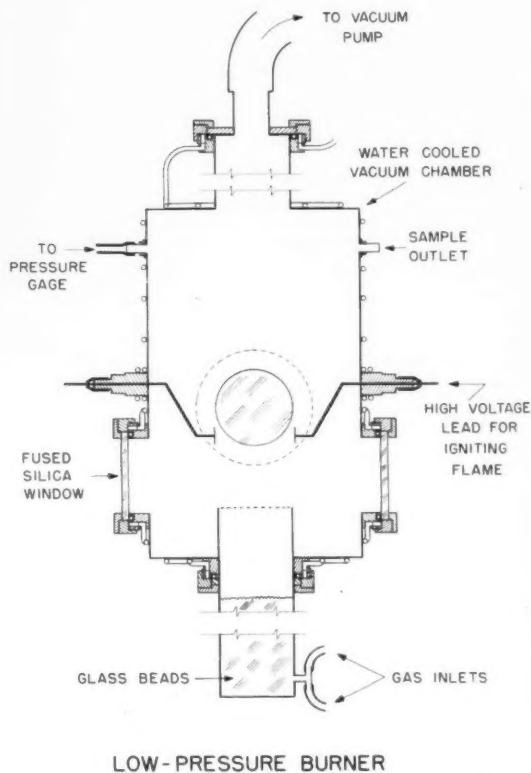
No Interaction

A second, quite different type of behavior is found in observing the OH radical emission spectrum in the reaction zone of an acetylene-oxygen flame.² Under certain conditions, observations reveal an abnormal rotational distribution in the electronically excited state, corresponding to a temperature approaching $10,000^{\circ}\text{K}$. However, the heat released in burning is sufficient to produce an equilibrium temperature no higher than $2,000^{\circ}\text{K}$. Results of this kind have been a puzzling contradiction for some time, since molecules that have such an excess of rotational energy are known

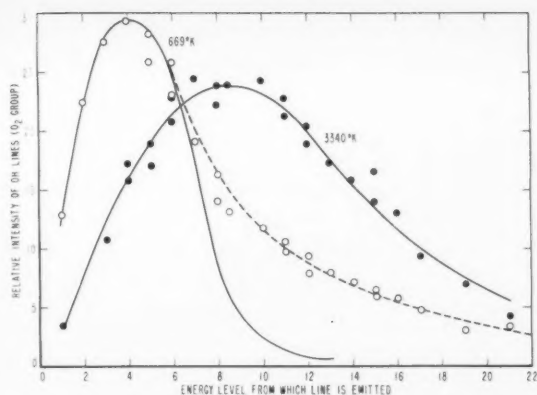
to lose this excess quickly in collisions with other gas molecules at a lower temperature. Why there seems to be no interaction between the abnormally populated rotational levels and other groups of energy levels in the system has not been understood.

This contradiction has now been resolved by the following considerations.^{3,4} The observed electronically excited OH molecules are originally formed with a large excess of rotational energy. If this excess is to show up in the spectrum, the molecules must radiate before they have undergone enough collisions with other molecules in the flame gases to bring their energy back to normal. A study of the fluorescence of these OH radicals in flames has shown that this electronic excitation energy is lost at almost every collision, so that the excited molecules do not live long enough in the flame to undergo the number of collisions—approximately 10—which are necessary to affect their rotational energy. Changing conditions in the flame cannot change the distribution unless they affect the excitation process itself.

In the experiments bearing on this point, an acetylene-oxygen flame, burning at a few thousandths of an atmosphere pressure, is illuminated by an intense source of light. This light is absorbed by the OH radicals in the burnt gas. If there were no deactivating collisions,



Schematic diagram showing the burner used to produce low-pressure flames for combustion studies.



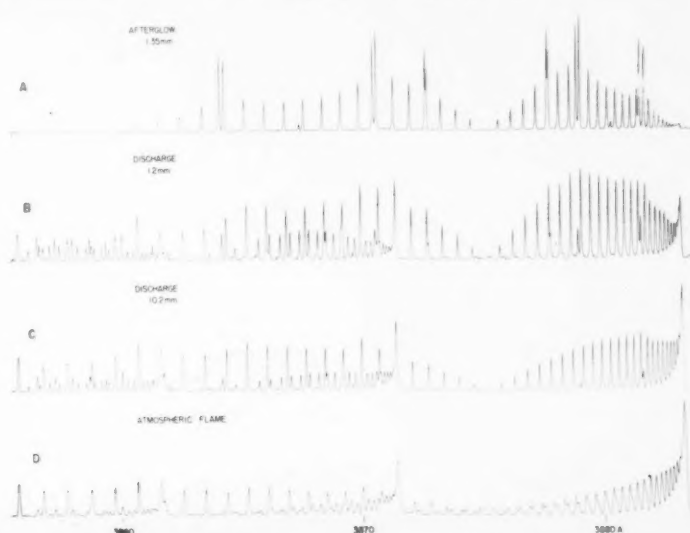
Distributions of OH emission line intensities in two electrical discharges. The agreement between calculated equilibrium curves (solid lines) and intensity measurements (experimental points) is shown.

all of the light absorbed would be reradiated as fluorescence. However, the fluorescence is found to be much weaker than expected. Therefore, most of the energy is lost in deactivating collisions which must have a high efficiency.

Partial Interaction

In the third possible type of behavior, the interaction between the several groups of energy levels is only partial, rather than being almost complete as in the hydrogen-oxygen flame, or nonexistent as in the abnormal OH spectrum mentioned above. In this intermediate case, the distribution of molecules in the accessible energy levels, and therefore the apparent temperature, is affected by molecular collisions. These collisions tend

Spectrum for CN radical emission. Changes in rotational temperature are seen most clearly in the region 3876 to 3884 Å, particularly in the lines at longest wavelength. The increase in vibrational temperature is indicated by the appearance of a second system of lines, arising from a higher vibrational level, in the region 3865 to 3871 Å. (A) Flame of atomic nitrogen and methyl chloride showing lines of abnormal intensity but with a low-rotational "temperature" near room temperature. (B and C) Discharge of benzene and nitrogen in an excess of argon showing low-rotational "temperature" and high-vibrational "temperature". (D) Acetylene-oxygen flame at atmospheric pressure showing rotational and vibrational equilibrium at the flame temperature.



to adjust the observed distribution to a form consistent with the distributions in other groups of energy levels and with the total energy content of the system. A theoretical treatment of certain processes of this type has recently been completed⁵ and relevant experimental data have been obtained from atomic flame and afterglow systems.^{6,7} Obtaining such data is complicated by the difficulty in distinguishing between changes in an observed distribution due to changes in the excitation process, and changes in the distribution due to collisions following excitation. With these problems in mind, studies are being made of the spectra emitted when nitrogen atoms react with nitrogen and oxygen molecules, and with simple organic molecules. Such studies have shown, for example, the rotational distribution of electronically excited CN radicals to be so strikingly affected by pressure increases that only at high pressures can a well-determined temperature be assigned to this group of energy levels.

¹Spectroscopic study of electronic flame temperatures and energy distributions, by H. P. Broida and K. E. Shuler, *J. Chem. Phys.* **27**, 933 (1957).

²Rotational temperatures of diluted flames, by W. R. Kane and H. P. Broida, *J. Chem. Phys.* **21**, 237 (1953).

³Fluorescence and average lifetime of OH²Σ⁺ in flames, by H. P. Broida, and T. Carrington, *J. Chem. Phys.* **23**, 2202 (1955).

⁴Electronic quenching of OH²Σ⁺ in flames, and its significance in the interpretation of rotational relaxation, by T. Carrington (to be submitted to *J. Chem. Phys.*).

⁵Studies on nonequilibrium rate process. I. The relaxation of a system of harmonic oscillators, by E. W. Montroll and K. E. Shuler, *J. Chem. Phys.* **26**, 454 (1957).

⁶Spectra of afterglows and discharges from nitrogen-oxygen mixtures, by U. H. Karzweg, A. M. Bass, and H. P. Broida, *J. Mol. Spec.* **1**, 184 (1957).

⁷Emission spectra from mixtures of atomic nitrogen and organic substances, by N. Kiess and H. P. Broida (to be delivered at Seventh International Symposium on Combustion, London, England, Aug. 1958).

High-Temperature Strain Gages

A PROGRAM aimed at improving existing strain gages and developing new types for high-temperature use is being carried on in three laboratories at the Bureau under the sponsorship of the Navy, Air Force, and Atomic Energy Commission. The work includes the improvement of ceramic-bonded gages, the development of strain-sensitive evaporated film gages, and the evaluation and calibration of presently used and proposed strain-measuring devices.

Strain gages are required to determine the suitability of structural materials for high-temperature applications. Such characteristics as strength, durability, and dimensional stability are of critical importance in aircraft engines, aircraft and missile airframes subject to aerodynamic heating, and certain types of nuclear reactors and reactor coolant systems. The measurement of strain in such structures not only provides the engineer with knowledge of the performance characteristics of the structure, but also gives information of value for improving the design. At present, gages are urgently needed to perform satisfactorily up to 1,000° F, and in the near future instruments for use up to 2,000° F will be in demand. These gages must be suitable for both static and dynamic tests on a wide range of different materials under both steady and transient temperature conditions. They should also be capable of measurements under field conditions.



Spot-welding strain-gage elements to a ceramic-coated asbestos backing.

Strains at or near room temperature are usually measured with paper-backed, wire-resistance gages of the SR-4 type. The gages are cemented to the structure with an organic resin. When a load is applied, the wire filament of each gage is deformed by approximately the same amount as the surface to which it is cemented. This deformation results in a change in electrical resistance of the wire filament. A knowledge of gage characteristics permits such changes to be converted to strain. The maximum temperature at which the paper-backed gage will operate is about 350° F. Above this temperature, the organic cements deteriorate and will no longer transmit the full amount of strain to the filament wire.



Top: Strain-sensitive film deposited directly on a non-conducting portion of a test strip. Bottom: Ceramic-coated asbestos-backed gage (filament-side down) bonded to test strip with ceramic cement.

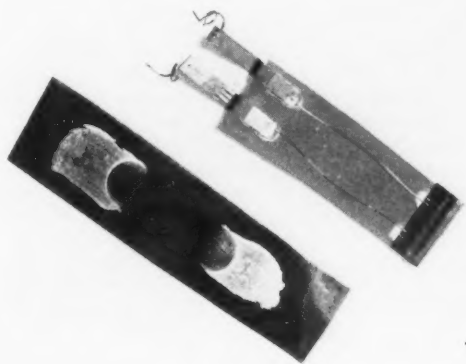
For high-temperature applications, the Bureau is attempting to solve this deterioration problem in two ways: by modifying the usual type of resistance strain gage through use of ceramics in place of organic materials; and by developing a gage which, in one form, eliminates cements by employing directly deposited conducting films instead of wires.

Ceramic-Bonded Gages

The work on ceramic-bonded gages has been underway in the enameled metals laboratory for the past three years, under the joint support of the Navy Bureau of Aeronautics, the Wright Air Development Center, and the Navy Bureau of Ships. From this investigation has resulted a new gage, called the 5B

design, which gives usable static-strain data up to 800° F. Developed by D. G. Moore and J. W. Pitts of the Bureau staff, this device is now commercially available.

The gage consists of two straight wires, 0.001 in. in diameter by 7/8 in. in length, welded to narrow strips of Inconel foil, 0.002 in. thick. The Inconel strips, which form the leads and shunt at the bottom of the gage, are crimped to a backing of asbestos paper previously coated with a thin layer of ceramic cement. When the gage is used, it is cemented to the surface of the test structure with the same ceramic cement. No precoating of the test surface is necessary. The wet cement does not completely flow out from under the Inconel strips when the gage is pressed into position; hence, there is no electrical shorting to ground. Measurements have shown that the filament wires are from 0.001 to 0.002 in. from the test surface after the gage has been applied.



Left: A strain-sensitive film evaporated on a foil foundation between electrical contacts. **Right:** The underside of a ceramic-coated asbestos-backed gage.

One advantage of the device is that it is completely prefabricated and therefore can be attached to many recessed areas of the test structure. Most commercial gages are mounted on a resin backing and this backing must be removed before the gage is imbedded in the ceramic cement. This requires considerable skill on the part of the operator. Such gages also require that a ceramic precoat be applied to the metal.

The maximum temperature at which the 5B gage is usable is limited by the decrease in resistance stability of the wires with temperature and by loss of resistivity in the ceramic cements. To extend the upper range of temperature, attention is being focused on cements that show higher electrical resistivity at high temperatures. A cement has recently been developed which has improved properties in the range 1,400° to 1,800° F. Ceramic coating of the alloy filament is also being investigated both to improve resistance stability of the wire and to aid the insulating cements in preventing electrical leakage to ground.



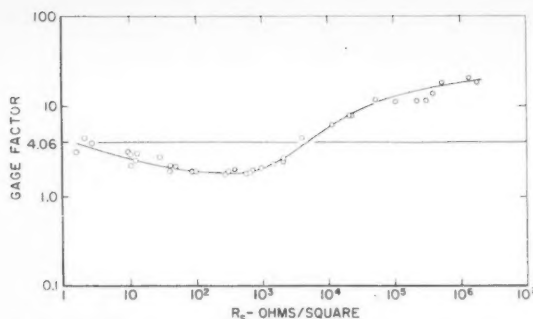
This apparatus for controlling the temperature of a vertically-strained specimen is used in the development of evaporated-film strain gages.

Strain-Sensitive Film Gages

Another approach to the development of a high-temperature strain gage is being made by A. Krinsky and his associates in the Bureau's Mechanical Instruments Section. The technique under consideration involves the use of deposited metal or alloy films in place of attached filaments as strain-gage elements. Investigations of the suitability of such films for use as high-temperature strain-gage elements are being carried out under a program sponsored by the Atomic Energy Commission.

In the proposed gage, the film will be deposited onto a thin nonconducting substrate that can be bonded to test surfaces. Alternatively the film may be deposited on an area of the test surface that has been rendered nonconductive, thus eliminating the use of cements or adhesives. Electrical contacts bonded to the test surface at either end of the film will permit strain measurements to be made.

In developing an evaporated film strain gage for use at temperatures from 1,000° to 2,000° F, several factors must be considered. Of prime importance is the choice of film material. The film must be strain-sensitive, capable of adhering to the test surface, and chemically and physically stable at high temperatures. The test surface must be electrically nonconducting. If gages are to be used within atomic reactors, a further restriction is placed on the choice of film and base material, since both must be unaffected by nuclear radiation.



Room temperature studies of palladium films used as strain gages. Experimental points show the gage factor as a function of specific resistance. The straight horizontal line indicates the bulk gage factor.

In studies of the resistance-strain properties of films evaporated from 22 metals, the Bureau found all films to be strain-sensitive. These tests were conducted on films deposited on suitable test strips by a vacuum evaporator. The foundations were an anodized aluminum strip coated with silicone resin and having silver contacts, and a porcelain-coated steel strip with platinum contacts. The test strip with its film gage was subjected to strain, and the resistance-strain relationship determined.

The strain-sensitivity or gage factor of the test films was found to depend strongly on specific resistance or thickness of the film; for the thinner films strain sensitivities several times those of the bulk material value were observed. The relationship between gage factor and specific resistance was similar in form for all metals tested. Further tests showed that the gage factor is a true property of the film, independent of contact, substrate, or atmosphere effects.

Although several of the elementary metals gave satisfactory resistance-strain curves at room temperature, at higher temperatures (starting at 200° F) resistance and gage factor were found to decrease. Time also had a deleterious effect. By electron microscope and electron and X-ray diffraction methods, these property changes were seen to result from changes in structure.

For these reasons, attention was turned to the development of an alloy that would retain its resistance properties at elevated temperatures and over long time intervals. Satisfactory results up to 750° F (the maximum temperature at which tests have been conducted) have been obtained in preliminary tests with an alloy film evaporated from a mixture of iron, aluminum, and titanium powders.

In addition to strain-sensitivity and durability, material suitability depends on factors such as resistance-strain stability, reversibility, and linearity. Some drift, hysteresis, and nonlinearity were noted in all resistance strain curves for elementary metals. However, the alloy appeared to be stable under all test conditions.

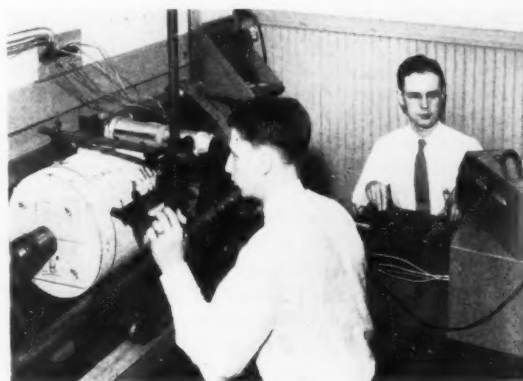
Before evaporated-film gages can be applied directly to structural test surfaces, some means of adequate bonding between contacts and insulating coatings must

be developed. Transferable film gages on foil foundations have been produced and found suitable for use with organic cements up to 450° F. However, loss in shear strength of the cement limits their use at this temperature. Ceramic cements do not as yet offer a solution to the problem as they do not cure properly when confined between the two nonporous surfaces.

Strain-Gage Evaluation

Since resistance strain gages cannot be individually calibrated prior to use, their usefulness depends upon the characteristics of the gages being accurately known. At elevated temperatures these gages often develop deficiencies which are not necessarily predictable, but which may be serious enough to limit the usefulness of the gages. To determine the performance characteristics of available gage types, to define the conditions under which meaningful measurements may be made with the gages, and to point out areas where further gage development is needed, an evaluation facility has been established at the Bureau under the sponsorship of the Navy and Air Force. Equipment and procedures designed by the Bureau's engineering mechanics laboratory under the direction of R. L. Bloss and C. H. Melton are now available for the comprehensive evaluation of gages at temperatures up to 1,000° F.¹ Some of these tests were carried out at temperatures up to 1,500° F. Tests include the determination of gage factor, resistance drift at elevated temperatures, resistance-temperature relationships, the effects of transient heating on resistance characteristics, and the effects of storing at various humidity levels.

In obtaining gage factor values, the resistance gage is attached to a uniform test strip in a region of controlled temperature. Change of gage resistance as the strip is strained is measured with a Wheatstone bridge circuit capable of detecting a relative change of resistance of 1 part per million. Strain to which the resistance gage is subjected is measured with an optical strain gage which can be read through a window provided in the temperature-controlling furnace. After



The evaluation of high-temperature strain gages includes a determination of gage factor at controlled temperature. The strain in a specimen within the furnace is indicated by an optical strain gage, read through window. The relative change of resistance is measured by a Wheatstone bridge (background).

corrections for temperature fluctuations, elapsed time, and instrument calibration are applied to the resistance-strain data, the gage factor is determined as the slope of the curve of relative change of resistance versus strain. The departure of the experimental points from a straight line shows the nonlinearity of gage response. Hysteresis and zero shift are also shown by data taken during increasing and decreasing loading.

The determination of change of gage resistance with time at elevated temperatures is necessary since such drift can cause serious error in static strain measurements. To determine drift, a gage attached to a stainless steel test strip is inserted into an oven preheated to the desired test temperature. When the test strip has come to the oven temperature, the apparent strain indicated by the gage is noted at frequent intervals for at least 1 hr. Such drift measurements have been made at temperatures up to 1,200° F.

To apply corrections for temperature fluctuations during gage factor and drift tests, temperature coefficients of resistance are required. These values have been obtained by causing the temperature of the gage to increase a few degrees and to decrease a similar amount while the resistance change is noted. The temperature coefficient can be obtained from simultaneous equations involving resistance change, temperature, and time. This method assumes that temperature coefficients and drift rates are constants and therefore independent of small temperature variations, of direction of temperature change, and of time. Since these assumptions may



Measuring resistance of high-temperature cements. Temperature of the cement, in the cylindrical furnace (upper left), is controlled by the variable autotransformer (left), and indicated on the instrument in foreground. Cement resistance is read from meter on shelf.

not always be valid, temperature coefficients are also determined as the slope of a line drawn tangent to the resistance-temperature curve at the temperature under consideration. Curves recorded during heating rates of about 5° F/sec serve as a basis for these determinations.

Because many of the uses for high-temperature strain gages involve transient heating, a study of the effect of rapid temperature changes on the performance characteristics of the gages is conducted. Gages are subjected to repeated heating and cooling between 80° F and 1,500° F at rates of about 60° F/sec. Two banks of radiant heaters provide the heat; an air stream from a centrifugal blower accomplishes the cooling. During such temperature cycles, the relative change of resistance of the gage is recorded as a function of the test strip temperature.

To determine the effects of storage at different humidities, gages in three conditions—as received from the manufacturer, imbedded in uncured cement, and imbedded in cement and cured—are placed in closed containers with saturated salt solutions that produce atmospheres with relative humidities of about 12, 55, and 93 percent. The resistance of each gage is then measured at suitable time intervals, and the change of resistance is taken as evidence of the detrimental effects due to storing.

No gage as yet developed is entirely suitable for use at elevated temperatures. However, work on the improvement of existing gages and the development of new gages promises to extend measurements into the high-temperature region. In the meantime, a knowledge of characteristics of presently available gages, as provided by the Bureau's evaluation center, may make possible meaningful strain measurements at intermediate temperatures.

¹ A facility for the evaluation of resistance strain gages at elevated temperatures, by R. L. Bloss, ASTM Symposium on Elevated Temperature Strain Gages, Special Tech. Pub.



Vacuum evaporator used to prepare evaporated-film strain gages. The substrate for the film is suspended directly above a boat containing the metal to be deposited. Here the powdered metal is being put in a tube leading to the boat.

Standard Samples of High-Temperature Alloys

THE Bureau is conducting a program on high-temperature standard samples at the request of the Navy Bureau of Aeronautics and the Wright Air Development Center. Standards for both spectrometric and chemical analyses are being prepared to cover the composition ranges of 17 commercial types of high-temperature alloys. Research on new and improved methods of analysis is also underway, to insure accurate analysis of the standards and optimum conditions for their application in other laboratories.

The Bureau now distributes over 550 standard samples of chemicals, metals, and other materials to industrial and research laboratories for use in instrument calibration and process control. However, recent advances in jet aircraft, rockets, and guided missiles have brought about a need for additional standard samples to aid in controlling the composition of alloys that can withstand the high temperatures encountered in these applications.

Such alloys are composed of heavy elements in a variety of compositions. The principal alloying elements are iron, nickel, and cobalt in various proportions, to which may be added lesser amounts of carbon, manganese, silicon, chromium, molybdenum, tungsten, niobium, titanium, and aluminum. In addition to the major alloying elements, the minor or trace elements present in the alloys, such as boron or zirconium, may have marked effects on their properties.

Control of composition of the individual alloys within narrow limits is important to their fabrication and successful end use. However, because of the complexity of the alloys, chemical analyses are difficult and time consuming. On the other hand, optical and X-ray spectrometric methods offer considerable promise for rapid analysis provided that suitable standard samples of known composition are available for calibration of

the spectrometers. The spectrometric standards now being prepared should meet this need.

Both the Department of Defense and industrial groups have assisted in selecting the types of alloys needed for standards and the priority with which they should be prepared. As the result of meetings which the Bureau held with interested groups on June 15, 1956, and November 19, 1957, a series of 23 spectrometric standards of high-temperature alloys was selected for issuance, and production was begun. The alloys and nominal compositions to be covered by the proposed spectrometric standards are listed in table 1. A chemical standard sample for Waspaloy is also being prepared in the form of chips. Its nominal composition is as follows: C, 0.07; Cr, 19.0; Ni, 58.0; Co, 14.0; Mo, 4.0; Ti, 3.0; Al, 1.25; Mn, 0.5; Si, 0.25; Zr, 0.07; and B, 0.004 percent.

Standards for six of the alloys in table 1 (Nos. 1, 5, 6, 7, 12, and 17) have been prepared at the Bureau by remelting and casting commercial heats under argon. The six standards have been further processed at the Naval Gun Factory by forging into rectangular slabs, removing the center and end sections, and fabricating the remaining sections into rods. After homogeneity tests, the standards will be analyzed chemically and certified as NBS standard samples. A similar procedure will be followed for the remaining standards except that melting and fabrication will be done largely through commercial facilities.

In connection with these standards, methods of X-ray and optical spectrometric analysis are being investigated to provide for rapid tests of homogeneity and for determination of composition, especially for the minor and trace constituents. Also, improved chemical methods are being developed for the analysis of the various types of high-temperature alloys listed in table 1.

TABLE 1. Proposed spectrometric standards of high-temperature alloys

Name of alloy	Nominal chemical composition, percent													Number of standards proposed
	C	Mn	Si	Cr	Ni	Co	Mo	W	Nb	Ti	Al	Fe	Other	
1. AISI 316 ^a	b 0.10	b 2.0	b 1.0	17	12	-----	2.5	-----	-----	-----	-----	balance	-----	1
2. A-286	.05	1.35	0.50	15	26	-----	1.25	-----	-----	2.0	-----	balance	0.3V	3
3. M-308	.04	-----	-----	13	33	-----	3.0	6.4	-----	2.0	0.35	balance	.20Zr	1
4. Incoloy 901	.05	0.5	.35	13	40	-----	6.0	-----	-----	2.5	.20	balance	-----	1
5. 16-25-6	b .08	1.35	.70	16	25	-----	6.0	-----	-----	-----	-----	50	-----	1
6. 19-9DL	.30	1.10	.60	19.0	9.0	-----	1.25	1.2	0.40	0.30	-----	.66	-----	1
7. N-155	.15	1.5	.5	21	20	20	3.0	2.5	1.0	-----	-----	balance	.15N	1
8. M-252	.15	0.50	.50	19	55	10.0	10.0	-----	-----	2.5	1.0	2.0	-----	1
9. GMR-235	.15	b .25	b .60	15.5	balance	-----	5.25	-----	-----	2.0	3.0	10	.06B	1
10. Hastelloy W	b .12	b 1.0	b 1.0	5.0	balance	b 2.5	24.5	-----	-----	-----	-----	5.5	b .6V	1
11. Hastelloy X	.15	-----	-----	22	45	-----	9	-----	-----	-----	-----	balance	-----	1
12. Inconel "X" 550	.04	0.7	0.4	15	73	-----	-----	-----	0.9	2.4	0.9	7	-----	1
13. Inco 713	.12	.15	.4	13	balance	-----	4.5	-----	2.25	0.6	6.0	1.0	-----	3
14. Waspaloy	.07	.7	.4	19	56	14	4.3	-----	-----	3.0	1.3	1	-----	3
15. S-816	.38	1.20	.40	20	20	balance	4.0	4.0	4.0	-----	-----	4	-----	1
16. L-605	.15	1.50	.50	20	10	balance	-----	15	-----	-----	-----	-----	-----	1
17. Nimonic 80A ^c	b .1	b 1.0	b 1.0	19.5	balance	b 2.0	-----	-----	-----	2.25	1.25	b 5.0	-----	1

^a Stainless steel.

^b Maximum.

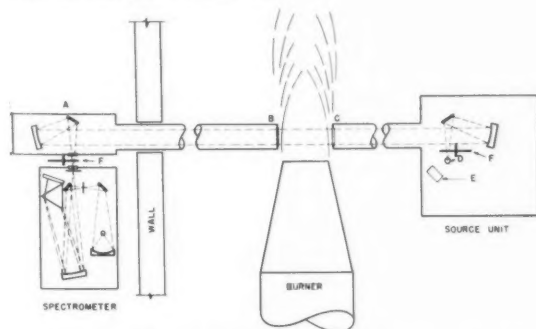
^c British alloy.

Emission and Absorption Studies of Hydrocarbon Combustion Products

THE Bureau has made a spectroscopic study of the infrared radiation from combustion gases under the sponsorship of the Wright Air Development Center. This work was done by G. A. Hornbeck and L. O. Olsen of the combustion controls laboratory to provide information that may make possible the measurement of jet-engine and gas-turbine exhaust temperatures. Ultimately such data should aid in the development of a flight-type radiation pyrometer to indicate and control the temperature of the hot gases that form the working medium for jet engines.

Under limited conditions, thermocouples can be used successfully to measure exhaust gas temperature and to operate the necessary control devices. However, in the afterburner of the newer aircraft engines, mass flows and exhaust temperatures are so high that no immersion-type instrument will survive in the stream. Temperature must therefore be determined from some property of the exhaust, measured by an instrument outside of the gas duct. Such an instrument could be a radiation pyrometer, which can "sense" gas temperature from the radiation characteristics of the hot products of combustion.

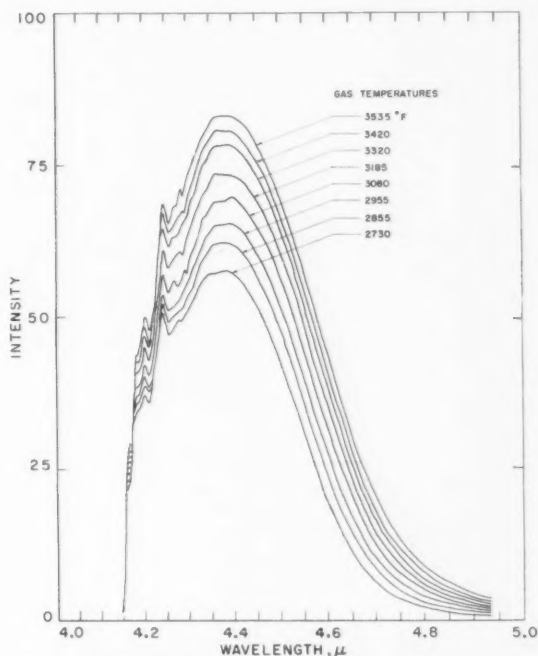
Radiance methods of temperature measurement depend upon Planck's distribution and Kirchhoff's radiation law. Planck's law relates the intensity of radiant energy from a blackbody to wavelength and temperature; Kirchhoff's law implies that the emissivity and absorptivity are equal at the same wavelength and temperature. Thus the two parameters, radiant energy and absorptivity, should be sufficient to define a gas temperature. However, measuring both absorptivity and intensity is often impractical. An alternate method for use with the jet engine pyrometer is suggested by the fact that certain spectral intervals exhibit fairly constant absorptivity even though temperature is varied. Thus in these regions, measurements of intensity only may be used to determine temperature on the basis of a previous intensity-temperature calibration for the spectral interval.



Apparatus used in study of jet-engine exhaust radiation. (A) Collimator; (B) and (C) sapphire windows; (D) globar; (E) radiation thermopile; and (F) choppers.

The present study of the emission and absorption characteristics of exhaust gases suggests the feasibility of such a temperature determination. Within the spectral range of interest, 1 to 5 μ , the optimum wavelength intervals were selected, and intensity variations under different conditions were investigated.

In this study, jet engine conditions were simulated by the use of a combustor producing a high-velocity exhaust stream in the temperature range from 800° to



Typical emission intensities for jet-engine exhaust gases in the spectral region 4.1 to 4.9 μ for temperatures from 2,730° to 3,535° F.

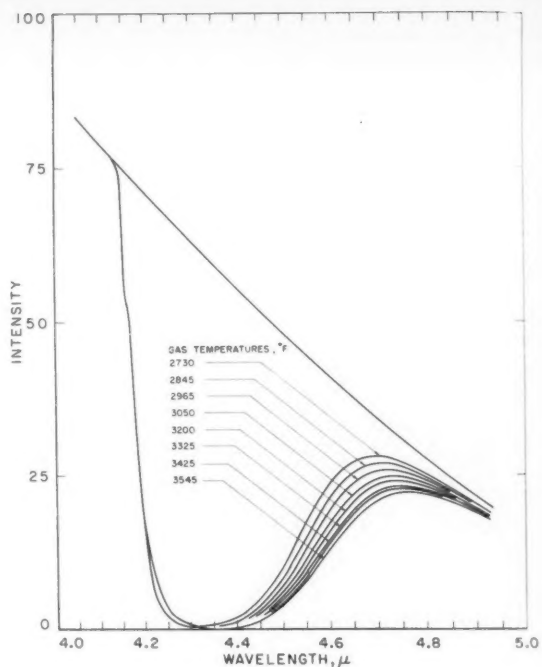
3,500° F. The spectra from the exhaust gases were obtained with the spectrometer and globar source located on opposite sides of the exhaust gas stream in order to permit both emission and absorption measurements. This arrangement necessitated the use of two mechanical choppers. When absorption measurements were made, only the chopper between the globar and the hot gas was active; when emission measurements were made, only the chopper between the gas stream and the spectrometer was active.

To locate the particular wavelength and spectral intervals where radiation is important, the entire range from 1 to 5 μ was scanned at various temperatures. From these observations, certain parts of this spectrum

were selected for more detailed investigation. First, radiation was monitored over the temperature range 1,000° to 3,600° F at 8 wavelengths: 1.34, 1.41, 1.84, 1.93, 2.52, 2.55, 2.70, and 4.39 μ . Then the radiation changes occurring within 2 intervals, 2.3 to 3.4 μ and 4.1 to 4.9 μ , were determined as a function of temperature. Within the first interval, radiation is emitted predominantly by water vapor, within the second mainly by carbon dioxide.

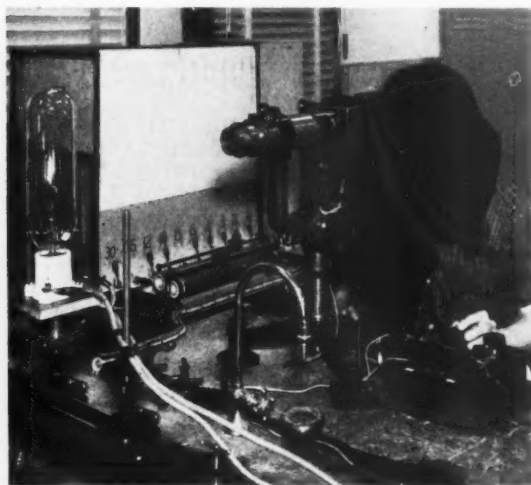
In the course of this work, data were also obtained on the effect of factors such as fuel-air ratio, mass flow, pressure, optimum time constant, optical path length, and background radiation. The results of these determinations indicate that gas-temperature monitoring would be most feasible in the spectral interval 4.1 to 4.9 μ since the requirement of large and fairly constant absorptivity is fulfilled in this region. As this radiation is from carbon dioxide molecules, a pyrometer might be designed around a carbon dioxide selective receiver. Evaluation of this type of receiver is now underway at the Bureau.

Typical absorption intensities for jet-engine exhaust gases in the spectral region 4.1 to 4.9 μ for temperatures of 2,730° and 3,545° F. Absorptivity is seen to be fairly constant over the wide temperature range.



International Comparison of High-Temperature Standards

PRECISE international comparisons have established that the optical pyrometer temperature scales of Canada, the United Kingdom, Germany, and the United States are in satisfactory agreement. Thus, within these countries, all measurements of high temperatures now have a common basis, and the results obtained in one laboratory should be comparable with those of another.



Above 1,063° C (the freezing point of gold) optical pyrometers are the standard instruments used to measure the brightness temperatures of incandescent bodies. This measurement consists of visual comparison of a portion of the radiation from the hot body with that emanating from an incandescent lamp filament contained in the pyrometer. The standard optical pyrometer is calibrated by determining the pyrometer lamp current which corresponds to a stated brightness temperature. This is done at 1,063° C by comparing its brightness with that from a hollow body (blackbody) surrounded by freezing gold. Higher temperatures are calculated from Planck's radiation equation utilizing measured brightness ratios.

Because the specially constructed optical pyrometers used as standards are not readily portable, it is not practical to send them abroad for international intercomparisons. However, it has been found that tungsten strip lamps, when used under rigidly controlled conditions, afford a means of reproducing brightness temperatures up to 2,300° C with a precision comparable to that attained with standard pyrometers.

Optical pyrometer (right), standard instrument for measuring temperatures above 1,063° C, is used to calibrate the brightness of a tungsten strip lamp (left). Scientist shields pyrometer during the reading to avoid error due to extraneous light.

Preparing for calibration of commercial optical pyrometer (center) using the standard pyrometer. Because the brightness temperature is influenced by the angle at which the observations are made, both instruments face the light source at the same angle.

In November 1955, both the National Bureau of Standards and the Physikalisch-Technische Bundesanstalt, the national standards laboratory of Germany, completed the calibration of two tungsten strip lamps, each laboratory comparing the lamps against its standard optical pyrometer. The results revealed a systematic difference, varying from 7° at $1,100^\circ\text{C}$ to 20° at $2,300^\circ\text{C}$, between corresponding values obtained at the two laboratories. Inasmuch as the National Bureau of Standards normally certifies the calibration of tungsten strip lamps to 3° at $1,100^\circ\text{C}$ and to 7° at $2,300^\circ\text{C}$, the discrepancy between the German and the American values caused some concern.

In 1956 a new primary calibration of the Bureau's standard pyrometer was performed, and the techniques used in the primary calibration and in the calibration of strip lamps were carefully investigated. Some minor changes in the lamp calibration values resulted, but these effects were very small compared to the discrepancy.

Arrangements were then made for an intercomparison of a group of calibrated tungsten strip lamps between the National Research Council of Canada and the National Bureau of Standards. Two of the Canadian lamps included in this exchange had previously received a routine calibration at the National Physical Laboratory in England in addition to the Canadian calibration. These intercomparisons among the three national laboratories, completed in April 1957, had a standard deviation of only 1.2°C for 90 points in the range from 800° to $2,200^\circ\text{C}$.

When these results were reported to the Physikalisch-Technische Bundesanstalt, the discrepancy was finally resolved. It was found that a new ceramic hollow body used as a blackbody in the German laboratory's primary pyrometer calibration had not completely fulfilled



blackbody conditions. This was recognized by the German laboratory shortly after the German intercomparisons, but the National Bureau of Standards did not receive this information until further tests had been completed. The resulting error in the pyrometer calibration had caused the apparent systematic error in the German-American intercomparison of tungsten strip lamps. Correction of this error has now brought about satisfactory agreement between the optical pyrometer portions of the International Temperature Scale as maintained in the four countries.

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Metallurgical Conference

MORE than 100 representatives of Government and industry attended the Bureau's annual metallurgical conference held at its Washington laboratories on April 29, 1958. Six technical papers, representative of current work in the metallurgical laboratories, were presented during the 1-day meeting.

The Metallurgical Conference has been an annual event since 1913, with the exception of the war years. Its purpose is to keep science and industry informed of the Bureau's progress in the study of the structure, behavior, and properties of metals and alloys. This research program is directed toward a better understanding of the properties of existing metals and the development of new or improved metals and alloys to meet new applications or to give better performance.

A. V. Astin, NBS Director, opened the conference. He briefly outlined the Metallurgy Division's role in helping the Bureau attain its scientific and technological objectives. Next on the program was James I. Hoffman, chief of the division, who announced the recent establishment of a metal physics laboratory, necessitated by the current emphasis on solid-state physics and the high-temperature properties of metals and alloys. Research has been initiated on such problems as nuclear magnetic resonance and mechanism and rate of growth of crystals from vapor.

In the first of the technical papers, W. J. Ambus discussed the reaction of atomic oxygen with metal single crystals. He related the results of an investigation on the rate and mechanism of reaction of copper with oxygen atoms. This study is part of a comprehensive program on free radical research. Preliminary experiments indicate that some attack occurred at 4°K and that the resulting product was a solid at room temperature. Mr. Ambus also described an optical method for measuring film thickness and optical constants on metal surfaces.

J. J. Park reported on the establishment of the equilibrium phases in the magnesium-zinc system. This study has clarified a previously existing uncertainty in the region of 45 to 85 weight percent of zinc, and has confirmed the existence of the Mg_7Zn_{23} , $MgZn$, and Mg_2Zn_3 phases.

R. L. Parker discussed the growth of metal crystals from their vapor and the role of screw dislocations in the mechanism of crystal growth. Very little quantitative information is available on the rate of growth of metal crystals under controlled conditions at low and well-defined supersaturation of metal vapor. Mr. Parker described experimental techniques for measuring these rates during the growth process.

A paper on the twinned epitaxy of copper on copper was given by T. H. Orem. X-ray diffraction studies

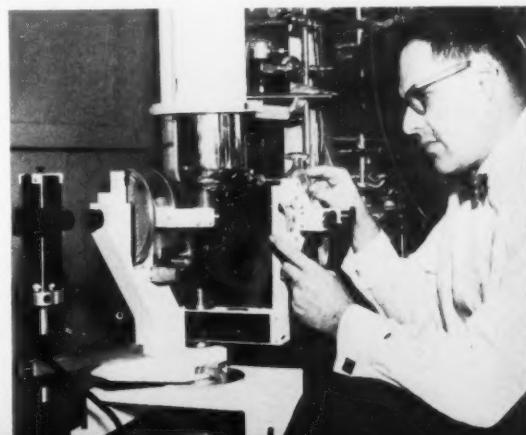
of copper electrodeposited on monocrystalline copper indicated that the microstructure of the deposit either duplicated the orientation of the basic metal or had both a continued and twinned relationship with it. Mr. Orem gave a detailed description of the growth and relationship of the deposit to the orientation of the underlying structure.

H. C. Burnett reported the results obtained in evaluating the torsional-fatigue properties of small-diameter high-carbon steel wire. The properties of the wire—processed from both commercial and vacuum-melted heats—in the initial conditions as cold drawn and as heat treated were evaluated in three different types of tests. Cold working, heat treating, and vacuum melting materially improved the wire's torsional fatigue properties. Results obtained with a recently developed torsion-fatigue machine and from fatigue tests on compression springs were in close agreement.

The progress made in evaluating materials for ultra-precise gage blocks was summarized by M. R. Meyerson. This work is part of a cooperative program¹ designed to produce gage blocks that are accurate to 1 part in ten million. An ideal material would be dimensionally stable, resistant to wear and to atmosphere and fingerprint corrosion, capable of acquiring an extremely fine surface finish, and have a coefficient of expansion similar to that of the material being gaged. Mr. Meyerson described the materials, processes, and treatments under investigation. He then indicated that quite promising preliminary results have been obtained in nitriding initially annealed blocks of 410 stainless steel. In tests thus far made on these blocks they appear to possess most of the properties required to produce gage blocks of the desired precision.

¹Because of the urgency of this work, appropriated funds available for the program have been supplemented by grants from the following industrial firms: Dearborn Gage Co., The DoAll Co., E. I. du Pont de Nemours & Co., Fonda Gage Co., Inc., General Electric Co., Greenfield Tap and Die Corp., Hughes Aircraft Co., International Business Machines Corp., Link Aviation, Inc., Pratt & Whitney Co., Inc., The Sheffield Corp., The Tait-Peirce Manufacturing Co., The Timken Roller Bearing Co., and the Van Keuren Co.

New equipment on display during the metallurgical conference included this ellipsometer recently acquired for studying corrosion reactions on the surfaces of metal single crystals. Instrument measures thickness of the thin films that form on a metal surface as it corrodes.



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U. S. DEPARTMENT OF COMMERCE
SINCLAIR WEEKS, *Secretary*
NATIONAL BUREAU OF STANDARDS
A. V. ASTIN, *Director*

July 1958 Issued Monthly Vol. 42, No. 7

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Publications of the National Bureau of Standards

Periodicals

Journal of Research of the National Bureau of Standards, Volume 60, No. 6, June 1958 (RP2867 to RP2876 incl.). 60 cents. Annual subscription \$4.00, \$1.25 additional for foreign mailing.

Technical News Bulletin, Volume 42, No. 6, June 1958. 10 cents.

Basic Radio Propagation Predictions for September 1958. Three months in advance. CRPL-D166. Issued June 1958. 10 cents. Annual subscription \$1.00, 25 cents additional for foreign mailing.

Research Papers

Journal of Research, Volume 60, No. 6, June 1958. 60 cents.

RP2867. Measurement of flame speeds by a nozzle burner method. Carl Halpern.

RP2868. Growth of preferentially oriented aluminum single crystals. Theodore H. Orem.

RP2869. Marginal performance of corrected ophthalmic lenses. Francis E. Washer and Walter R. Darling.

RP2870. Enthalpy and heat capacity from 0° to 900° C of three nickel-chromium-iron alloys of different carbon contents. Thomas B. Douglas and Ann W. Harman.

RP2871. Relative strengths of forty aromatic carboxylic acids in benzene at 25° C. Marion Maclean Davis and Hannah B. Hetzer.

RP2872. Shape of the liquidus surface as a criterion of stable glass formation. Edgar H. Hamilton and Given W. Cleek.

RP2873. Twinned epitaxy of copper on copper. Theodore H. Orem.

RP2874. Dielectric constant of deuterium oxide. Cyrus G. Malmberg.

RP2875. Additional abscissas and weights for Gaussian quadratures of high order: Values for $n=64, 80$, and 96 . Philip Davis and Philip Rabinowitz.

RP2876. Mass spectra of aromatic hydrocarbons filtered from smoky air. Fred L. Mohler, Paul Bradt, and Vernon H. Dibeler.

Index to volume 60 (January to June 1958, Research Papers RP2815 to RP2876).

Circulars

C460. (Supplement). Supplementary list of publications of the National Bureau of Standards July 1, 1947 to June 30, 1957. \$1.50.

C467. Vol. III. Atomic energy levels, as derived from the analyses of optical spectra. Charlotte E. Moore. \$2.50.

Applied Mathematics Series

AMS52. Integrals of airy functions. 25 cents.

Publications in Other Journals

The utility of meteor bursts for intermittent radio communication. G. F. Montgomery and G. R. Sugar. Proc. IRE 45, No. 12, 1684-1693 (Dec. 1957).

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Publications for which a price is indicated are available only from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. (foreign postage, one-fourth additional). The three NBS periodicals are available on a 1-, 2-, or 3-year subscription basis, although no reduction in rates can be made. Reprints from outside journals and the NBS Journal of Research may often be obtained directly from the authors.

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